UAH

GLASS SAMPLE CHARACTERIZATION

FINAL REPORT

Contract NAS8-36955
Delivery Order #4

Prepared for:
MARSHALL SPACE FLIGHT CENTER
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Prepared by:
Center for Applied Optics
The University of Alabama in Huntsville
Huntsville, Alabama 35899

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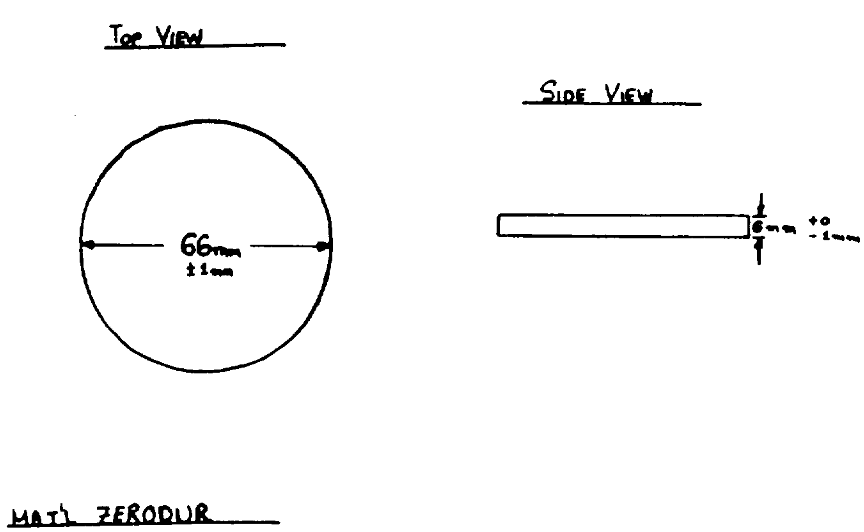
During the past year many glass samples were prepared using the material Zerodur to ascertain the strength under a variety of surface conditions which are expected to be encountered in the process of fabricating the AXAF mirrors. During the past year (1988-1989) 350 Zerodur samples were delivered to the material and processing laboratory at MSFC. Various equipment had to be used to analyze these surfaces and to ensure specifications are being met. Some of the equipment used were a good analytical weighing scale, Talystep, and a Zygo interferometer.

Mr. Khanijow also visited Lawrence Livermore and discussions were held in the area of analyzing sub-surface damage.

A lot of this will continue on through 1989-1990. Our task at MSFC was broken into two parts for 1988-1989. The primary effort was to prepare glass samples (material Zerodur) and perform a series of steps, such as cutting, surface generating, grinding, polishing and acid etching. These samples were ultimately used in a four-point bend test and a double ring break test. The final dimension of these samples was 6.6 cm diameter x 0.6 cm thick to +0/-1 mm (see Fig. 1-1).

FIGURE 1-1



Task I

The steps used were to cut and shape glass samples to size, after which we had to place these samples, a lot of ten at a time, onto the surface grinder. Since these samples were first cut with a diamond saw we had to place them onto the surface grinder to make both sides flat and parallel. To achieve this and at the same time to be consistent with the requirements for AXAF we had to use a 60/80 resin bonded wheel with 75% concentration. Approximate removal was .008" of material to ensure that most of the subsurface damage was removed and to achieve flatness. Upon completion of this task we then had to use a 230/270 metal bonded wheel and grind off at least .006" of material. For each step we had to be sure sufficient material was removed such that the damage produced by the previous process was eliminated. Our goal was to remove any residual damage produced during sawing or grinding.

A total of 175 samples were delivered, of which 12 samples from various batches were kept aside to be tested on the Talystep at UAH (see Fig. 1-2 charts 1 through 9). The Talystep has a vertical resolution better than 5Å. It provided simple, direct measurements and produced permanent graphical recordings step height and surface texture. These samples will be going through a fabrication process "wedge polishing" in 1989-1990 to continue our studies in subsurface damage.

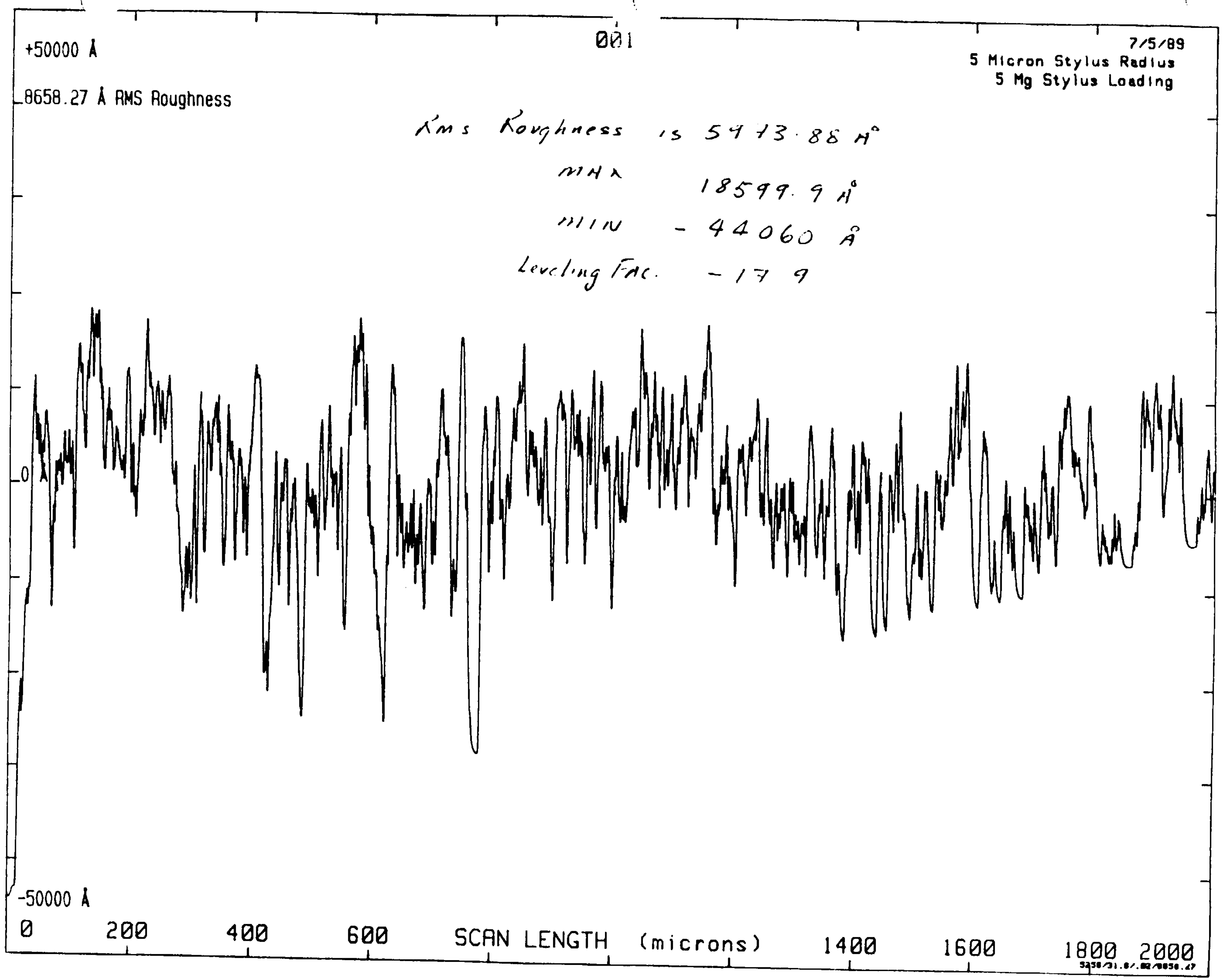
Task II - Acid Etching

The samples are acid etched to try to eliminate subsurface damage and stresses caused by surface grinding. In this case it was determined that 100 μm of material is to be removed in etching. For samples of this size (6.6 cm diameter x 0.6 cm thick) that translates to approximately 0.5 gm of material.

The surfaces have many microscopic cracks and crevices, so when it is first exposed to the etching agent the removal rate is highest. As the acid eats away the removal rate decreases and eventually levels off to a constant rate. In our first experiment we wanted to find out exactly when the removal rate levels off.

The etching solution consisted of water (90%) and hydrofluoric acid (10%). The sample was a 6.6 cm diameter by 0.6 cm thick disk

FIGURE 1 2



001
+50000 Å
8658.27 Å RMS Roughness
7/5/89
5 Micron Stylus Radius
5 Mg Stylus Loading
Rms Roughness is 5473.88 A°
MAX 18599.9 A°
MIN - 44060 Å
Leveling Fac. - 17 9
0 Å
-50000 Å
0
200
400
600
SCAN LENGTH (microns)
1400
1600
1800
2000

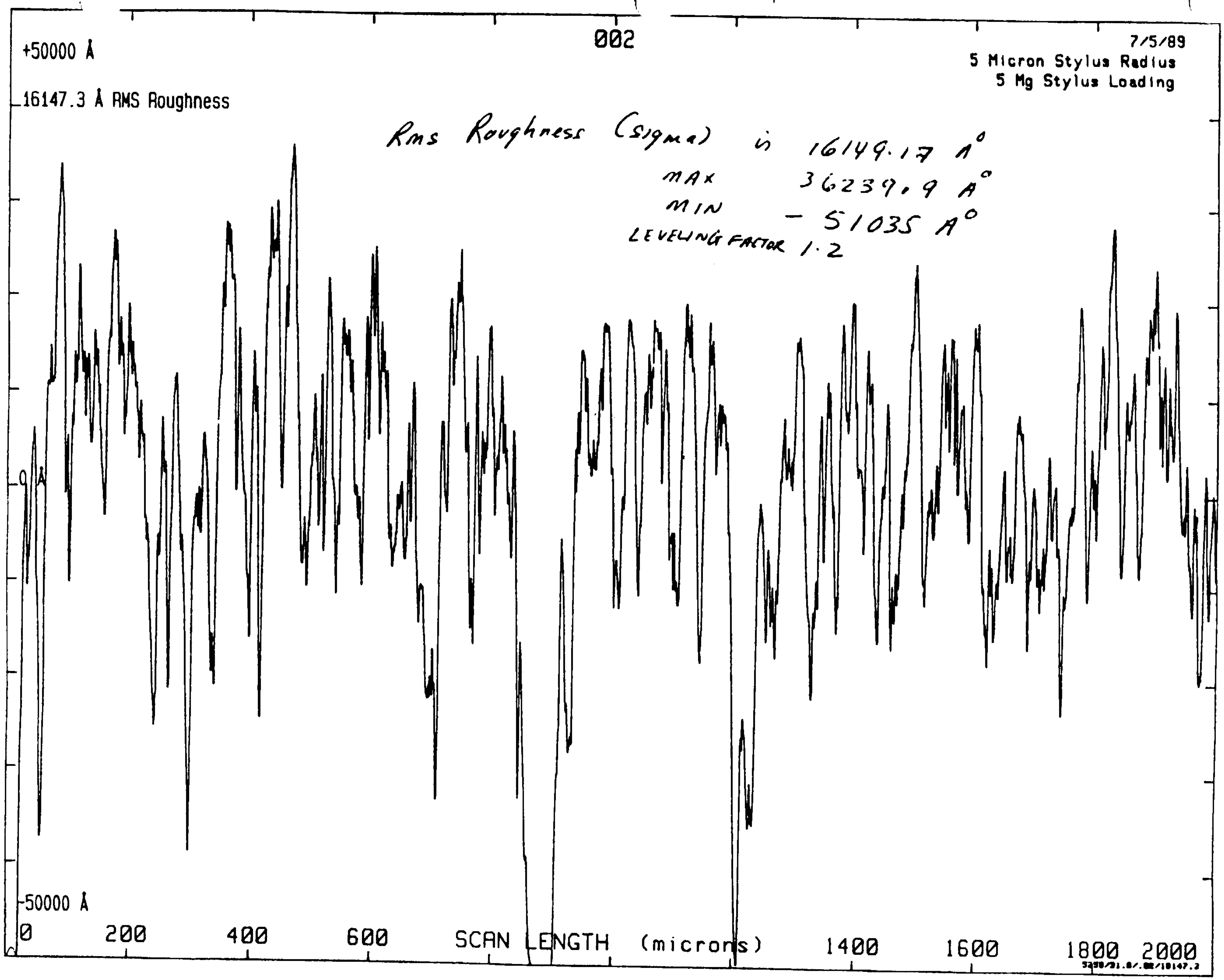

002
+50000 Å
16147.3 Å RMS Roughness
7/5/89
5 Micron Stylus Radius
5 Mg Stylus Loading
Rms Roughness (sigma) is 16149.17 A°
MAX 36239.9 A°
MIN - 51035 A°
LEVELING FACTOR 1.2
0 Å
-50000 Å
0
200
400
600
SCAN LENGTH (microns)
1400
1600
1800
2000

FIGURE 1

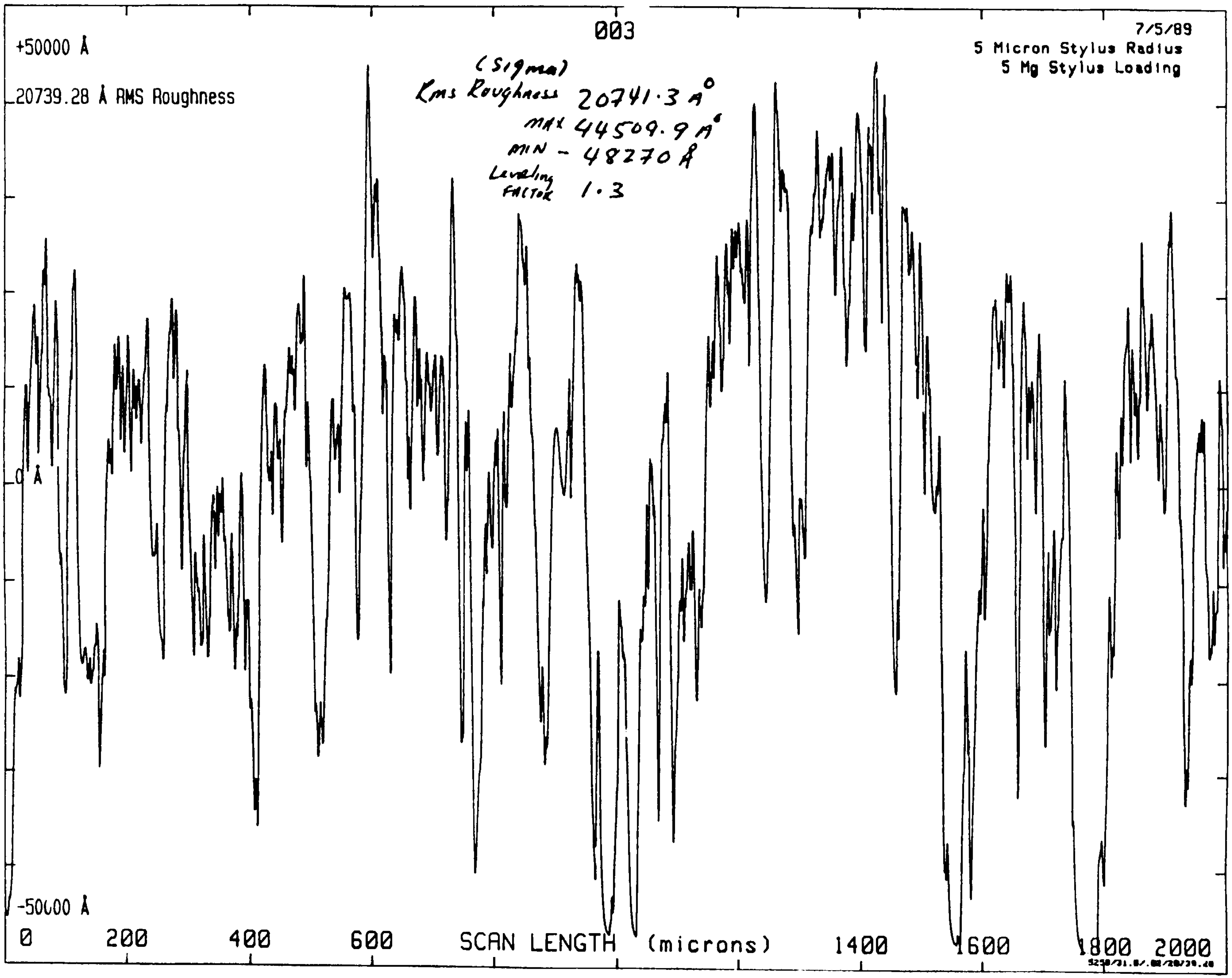
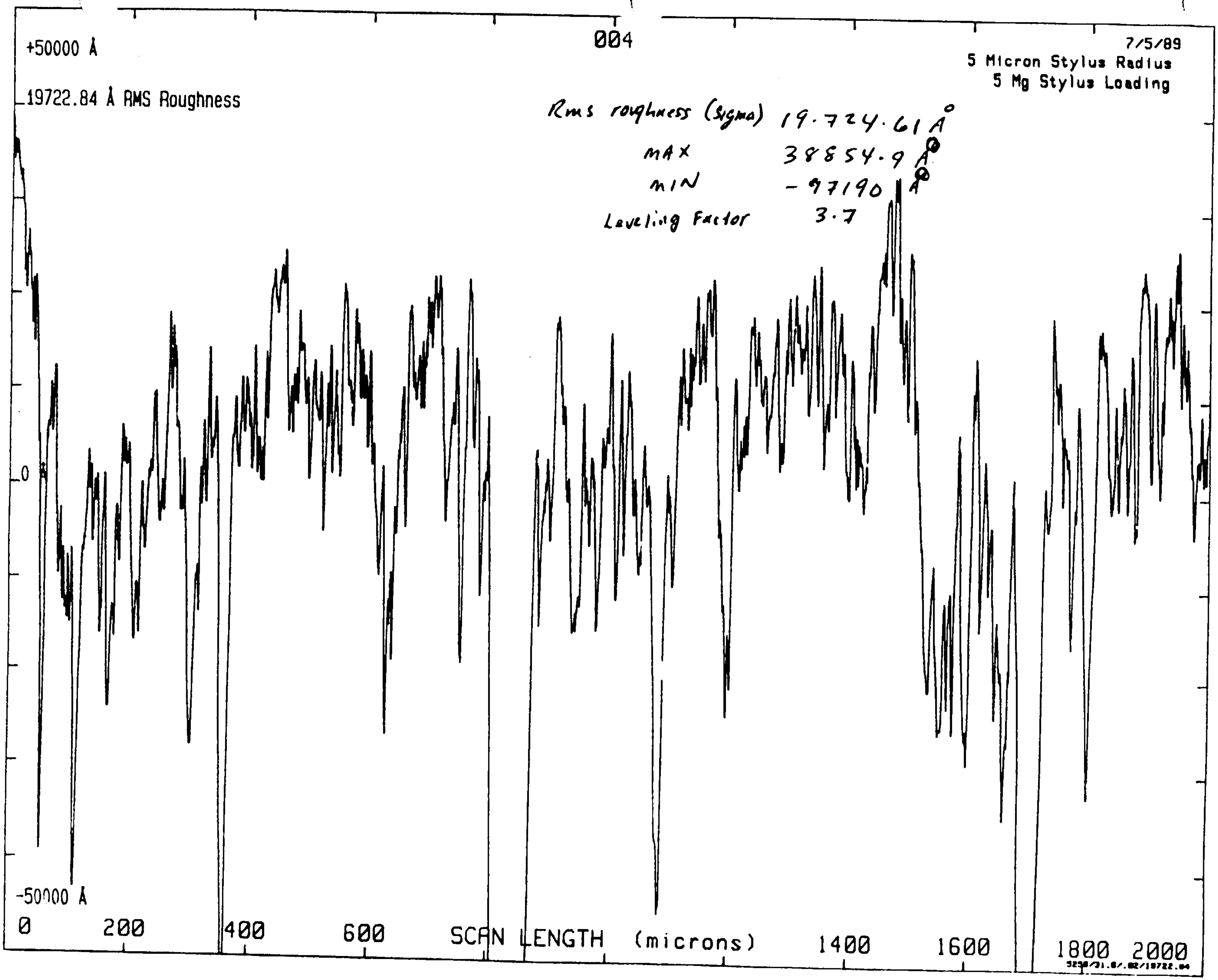
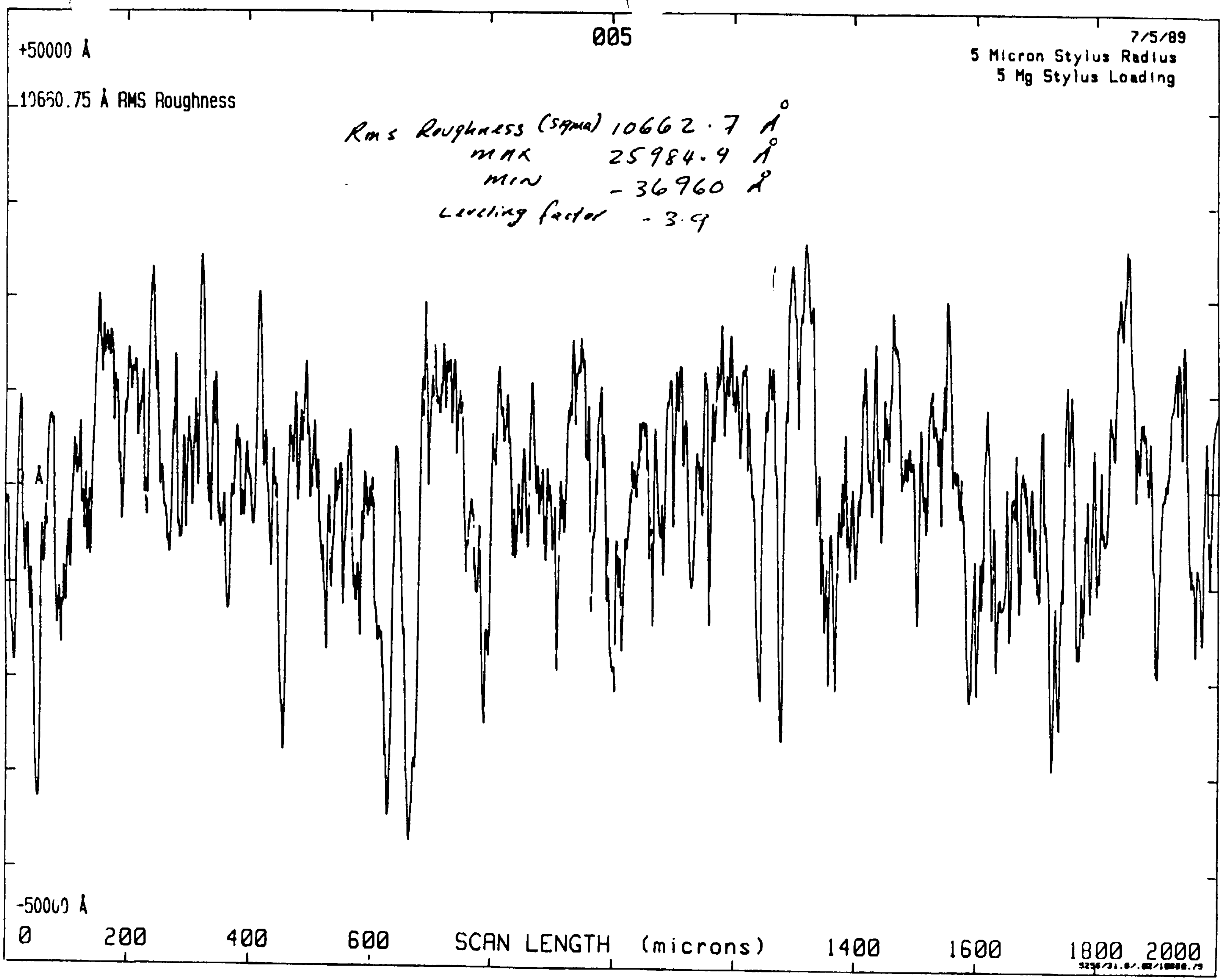
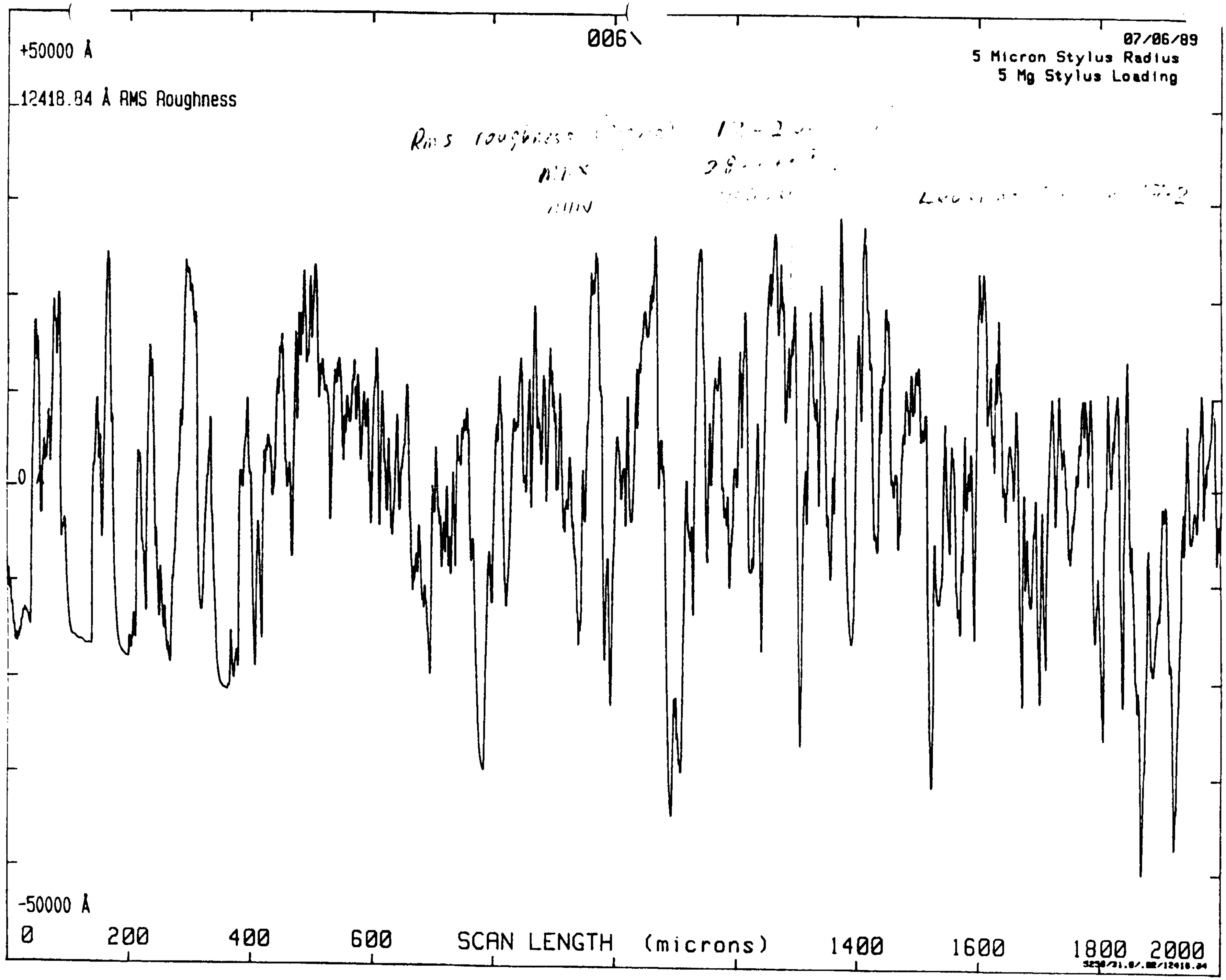

003
+50000 Å
20739.28 Å RMS Roughness
(Sigma)
Rms Roughness 20741.3 Å
MAX 44509.9 Å
MIN - 48270 Å
Leveling FACTOR 1.3
7/5/89
5 Micron Stylus Radius
5 Mg Stylus Loading
0 Å
-50000 Å
0
200
400
600
1400
1600
1800
2000
SCAN LENGTH (microns)

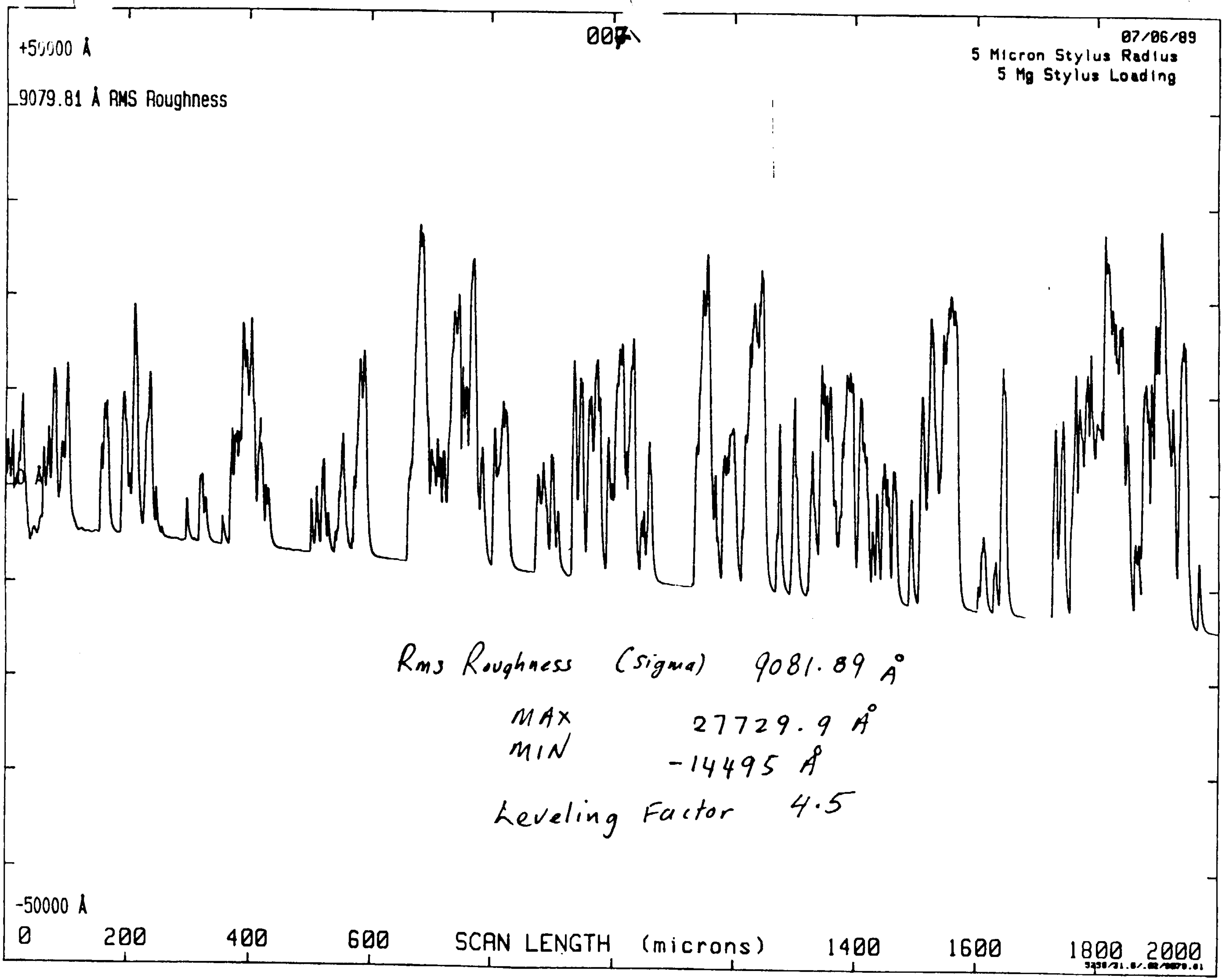
FIGURE 1-4



+50000 Å
19722.84 Å RMS Roughness
004
7/5/89
5 Micron Stylus Radius
5 Mg Stylus Loading
Rms roughness (sigma) 19.724.61 Å
MAX 38854.9 Å
MIN -97190 Å
Leveling Factor 3.7
0
-50000 Å
0
200
400
600
SCAN LENGTH (microns)
1400
1600
1800
2000


005
+50000 Å
10650.75 Å RMS Roughness
7/5/89
5 Micron Stylus Radius
5 Mg Stylus Loading
Rms Roughness (sigma) 10662.7 Å
MAX 25984.9 Å
MIN -36960 Å
Leveling factor -3.9
Å
-50000 Å
0
200
400
600
1400
1600
1800
2000
SCAN LENGTH (microns)


006
+50000 Å
12418.84 Å RMS Roughness
07/06/89
5 Micron Stylus Radius
5 Mg Stylus Loading
0
-50000 Å
0
200
400
600
SCAN LENGTH (microns)
1400
1600
1800
2000


+50000 Å
9079.81 Å RMS Roughness
007
07/06/89
5 Micron Stylus Radius
5 Mg Stylus Loading
Rms Roughness (Sigma) 9081.89 Å
MAX 27729.9 Å
MIN -14495 Å
Leveling Factor 4.5
-50000 Å
0
200
400
600
SCAN LENGTH (microns)
1400
1600
1800
2000

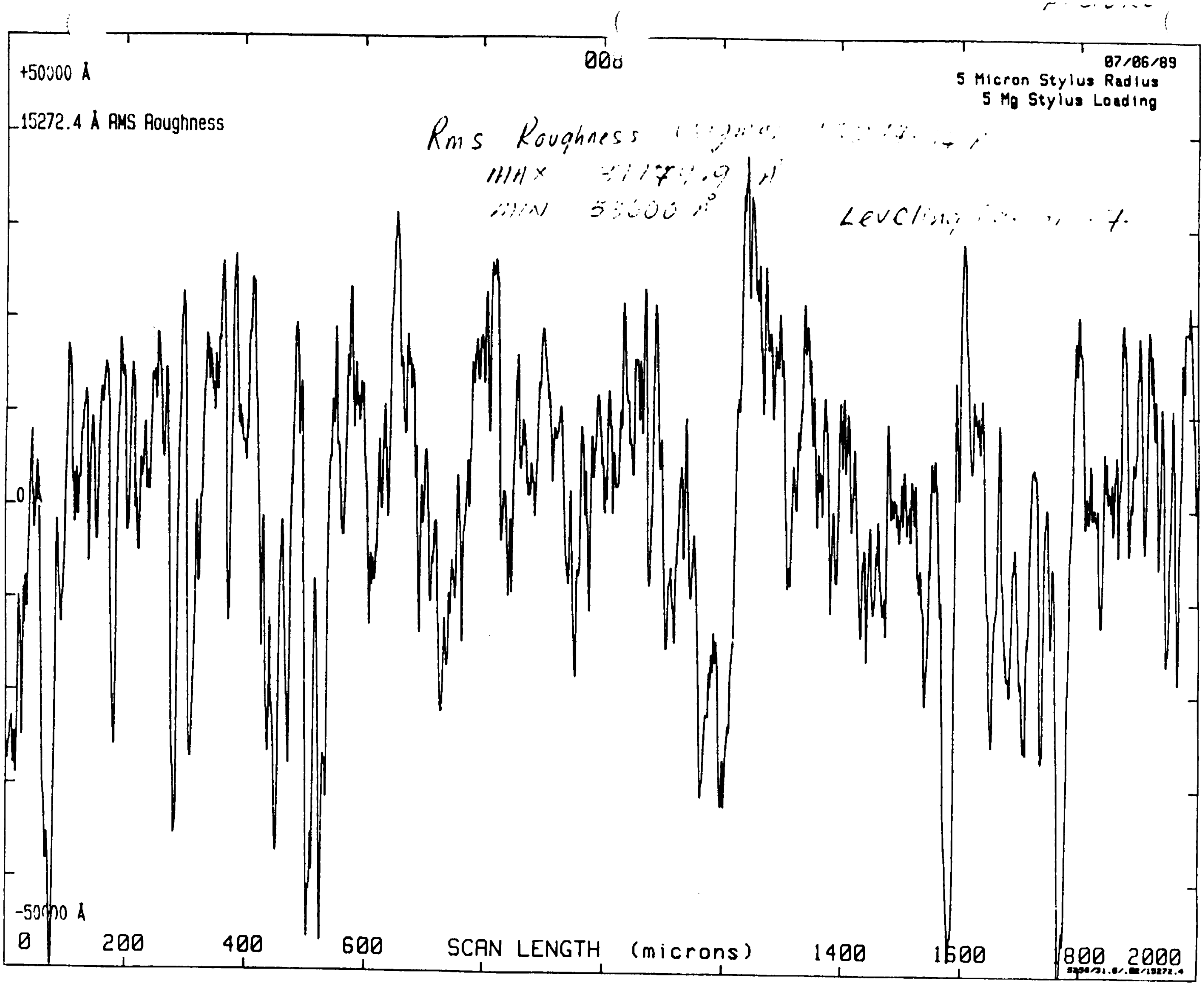


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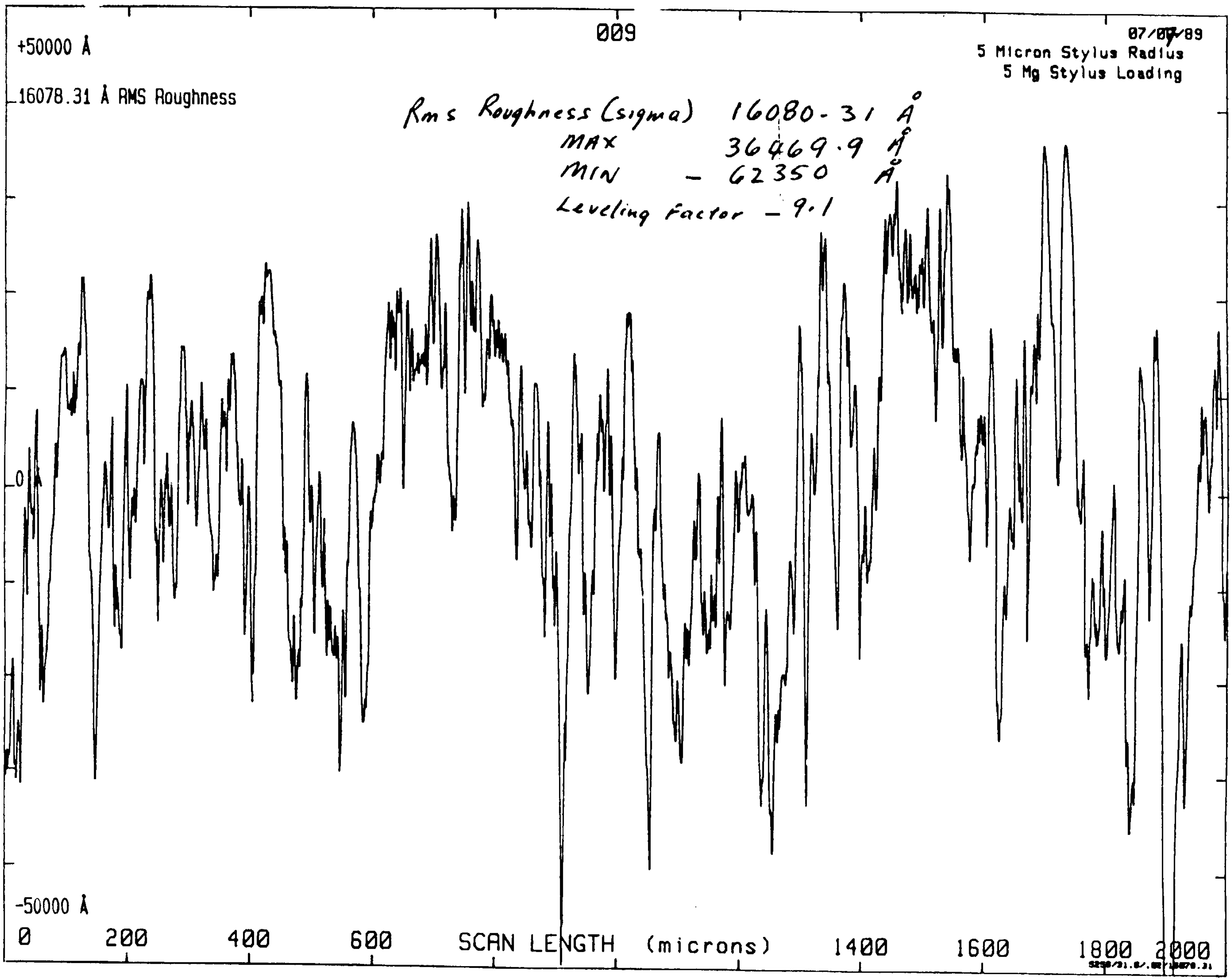
FIGURE - 2


+50000 Å
16078.31 Å RMS Roughness
009
07/07/89
5 Micron Stylus Radius
5 Mg Stylus Loading
Rms Roughness (sigma) 16080.31 Å
MAX 36469.9 Å
MIN - 62350 Å
Leveling Factor - 9.1
-50000 Å
0
200
400
600
SCAN LENGTH (microns)
1400
1600
1800
2000

of Zerodur with the edge and back side coarse-ground and the front face fine-grind finished with a 230/270 wheel. The sample was first weighed to 0.001 gm and then placed in the acid solution for 60 minutes in two minute intervals. The sample was weighed between each interval. When we plot removal rate versus time, we can see that the removal rate is highest during the first two minutes after which it quickly drops and begins to level off. Within 30 minutes the removal rate is almost constant (see Fig. 1-3). Also, at this time 0.505 grams of material has been removed. If all the subsurface damage has been removed we would expect the removal rate to begin to level off at this point. We should note here that time should not be a gage when etching many samples. It is best to use weight as a gage to ensure proper amount of material removed. In etching batches of samples, 10 pieces at a time, we had to extend etching time to as long as 75 minutes to remove 0.5 grams of material per sample.

The next part of the experiment was performed to compare removal rate to surface finish. Of course we would expect the removal rate to be higher with a coarser surface as there is more glass exposed to the etching agent, deeper cracks and crevices.

Two samples were prepared for this experiment. both were prepared similar to the previous one, fine-ground on one side with the 230/270 wheel. One sample was then waxed on the back side and edge so that only the fine-ground surface remained exposed. Since the hydrofluoric acid does not attack the wax, only the exposed surface would be etched. The second sample would be fully exposed. The samples were etched in two-minute intervals for the first 30 minutes and in five-minute intervals for the next 60 minutes for a total of 90 minutes of etching. The samples were weighed before starting and between each interval. The waxed sample was weighed before and after applying the wax.

Graphing removal rate (mg/min) versus time reveals, as expected, the removal rate for the unwaxed sample is higher since there is more surface area exposed (see Fig. 1-4). If we plot removal rate per unit area per minute versus time we can now better compare the two (see Fig. 1-5). The unwaxed sample still has a slightly higher removal rate because more than half its surface is

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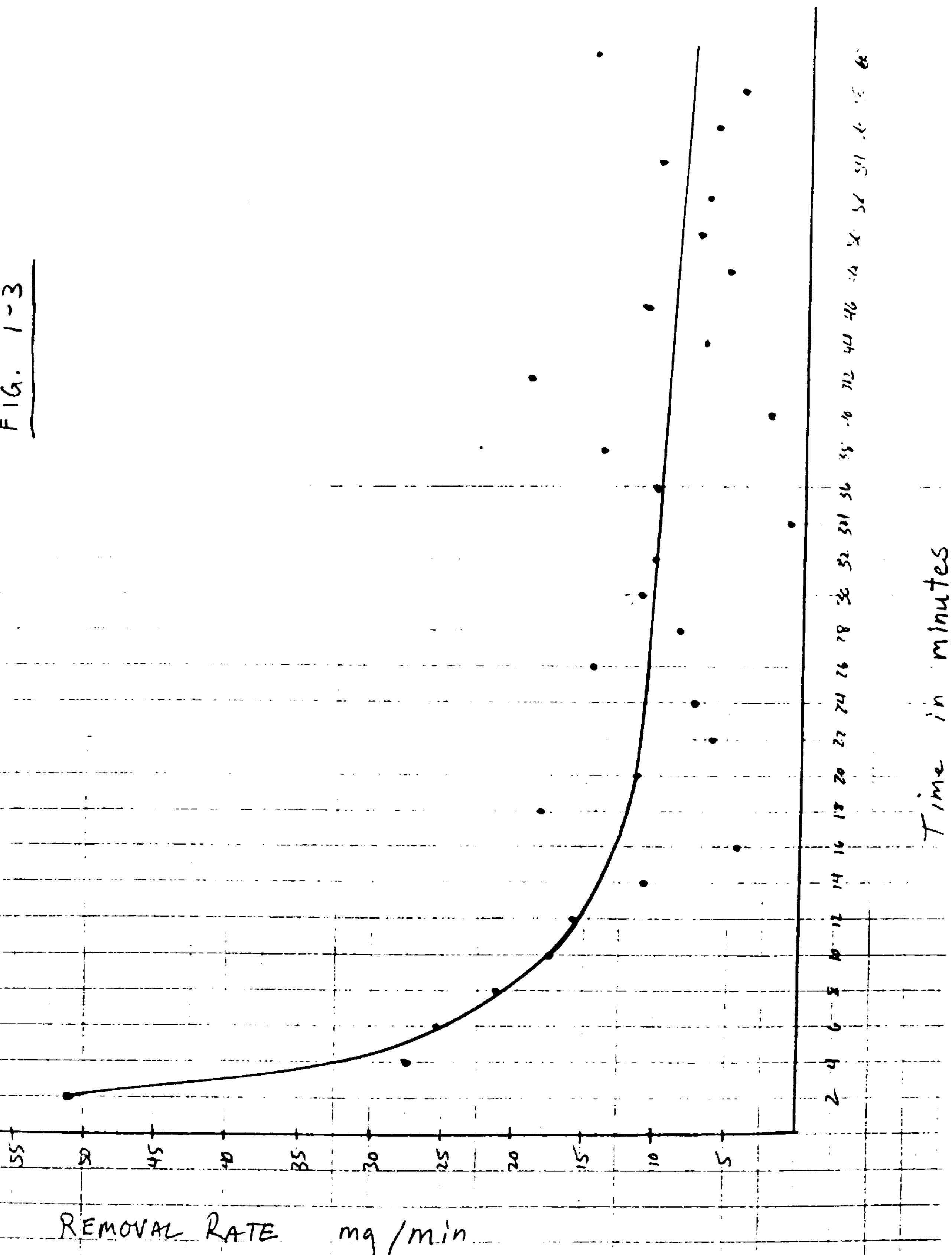

FIG. 1-3

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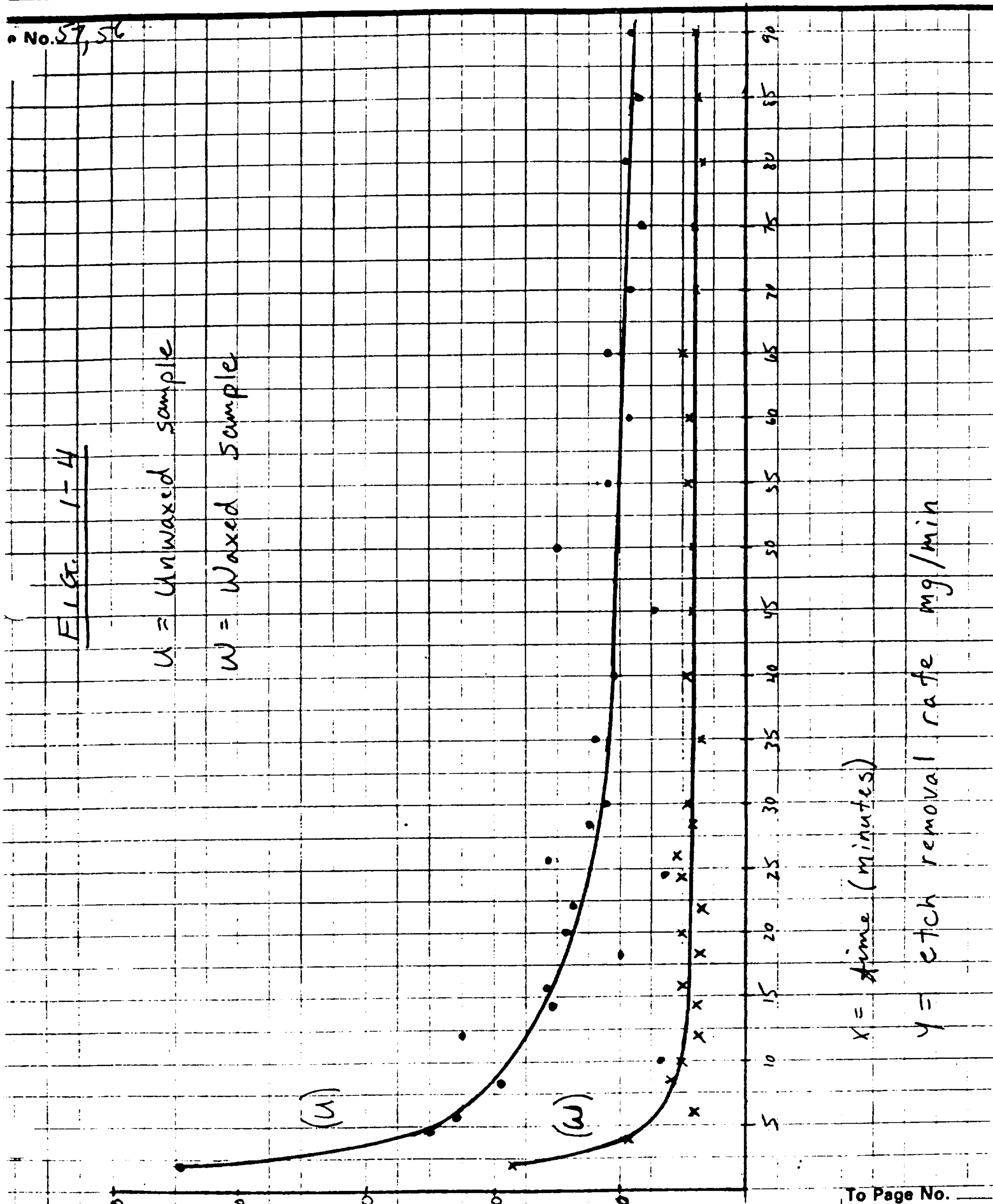

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Date

Invented by

Date

Recorded by

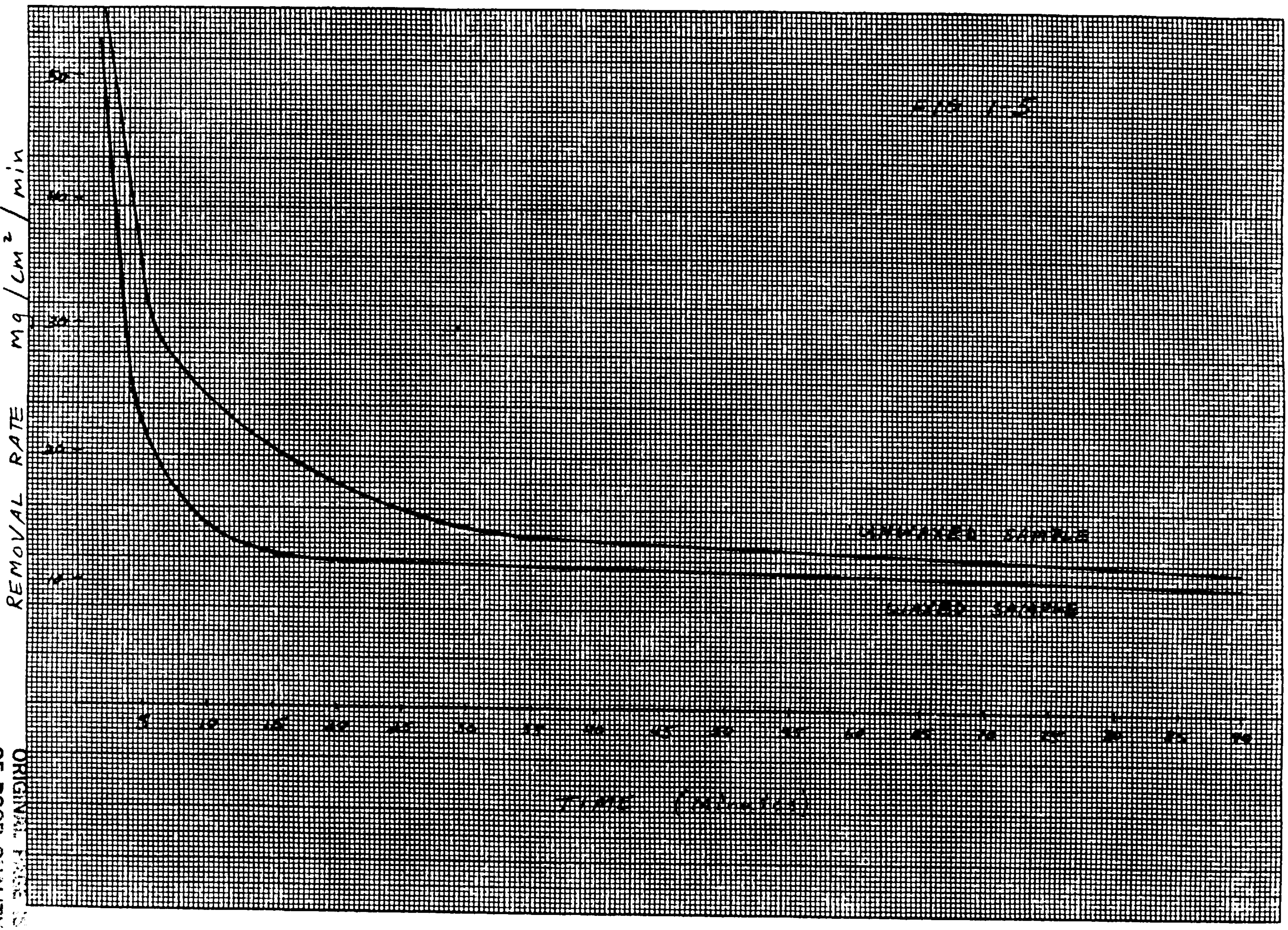
FIG 1-5
UNWAXED SAMPLE
WAXED SAMPLE
REMOVAL RATE mg/cm² / min
10
20
30
40
50
5
10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
TIME (minutes)

coarser. We can also see that the removal rate for the waxed sample (with the fine-ground surface) drops off slightly faster and levels off faster than the rate for the other sample with the rougher surface.

From these curves we will be picking various etch times for additional samples to investigate the effect of etch time on material strength.